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Jewellery — Determination of precious metals in 999 $^{0}/_{00}$ gold, platinum and palladium jewellery alloys — Difference method using ICP-OES

Joaillerie, bijouterie — Dosage des métaux précieux dans les alliages d'or, de platine et de palladium 999 $^{0}/_{00}$ pour la joaillerie, bijouterie — Méthode de la différence utilisant l'ICP-OES

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 174, *Jewellery*.

This second edition cancels and replaces the first edition (ISO 15093:2008), which has been technically revised with following changes:

— change in Clause 1 that this method is the referee method;

— addition of a warning in Clause 7 that suitable health and safety procedures should be followed;

— change of calibration solutions in 7.2 ;

— addition of an alternative wavelength for gold in Table A.1;

— editorial revision of this International Standard.

Introduction

The following definitions apply in understanding how to implement an ISO International Standard and other normative ISO deliverables (TS, PAS, IWA).

— “shall” indicates a requirement;

— “should” indicates a recommendation;

— “may” is used to indicate that something is permitted;

— “can” is used to indicate that something is possible, for example, that an organization or individual is able to do something.

ISO/IEC Directives, Part 2 (sixth edition, 2011), 3.3.1 defines a requirement as an “expression in the content of a document conveying criteria to be fulfilled if compliance with the document is to be claimed and from which no deviation is permitted.”

ISO/IEC Directives, Part 2 (sixth edition, 2011), 3.3.2 defines a recommendation as an “expression in the content of a document conveying that among several possibilities one is recommended as particularly suitable, without mentioning or excluding others, or that a certain course of action is preferred but not necessarily required, or that (in the negative form) a certain possibility or course of action is deprecated but not prohibited.”

Jewellery — Determination of precious metals in 999 $^{0}/_{00}$ gold, platinum and palladium jewellery alloys — Difference method using ICP-OES

1 Scope

This International Standard specifies an analytical procedure for the determination of either platinum in platinum jewellery alloys, gold in gold jewellery alloys, or palladium in palladium jewellery alloys, with a nominal content of each precious metal of 999 ‰ (parts per thousand) by measuring specific elements. (See Tables A.1, A.2, and A.3.)

This International Standard specifies a method intended to be used as the recommended method for the determination of fineness in 999 ‰ alloys covered by ISO 9202.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11596, *Jewellery — Sampling of precious metal alloys for and in jewellery and associated products*

3 Principle

The samples of the precious metal alloy are weighed and dissolved in aqua regia to prepare a 10 g/l solution. The impurities are determined by ICP-OES, and the precious metals content is obtained by subtraction of the total content of impurities in the sample from 1 000 ‰.

4 Sampling

The sampling procedure shall be performed in accordance with ISO 11596.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Hydrochloric acid (HCl), approximately 30 % to 37 % HCl (mass fraction).

5.2 Nitric acid (HNO_3), approximately 65 % to 70 % HNO_3 (mass fraction).

5.3 Aqua regia (should be prepared just before use).

Mix three volumes of hydrochloric acid (5.1) and one volume of nitric acid (5.2).

5.4 Acid stock solution (may contain both chlorides and nitrates), all relevant elements (100 mg/l each) in 1 mol/l HCl (5.1) and 1 mol/l HNO_3 (5.2).

5.5 Reference materials: gold, platinum, or palladium, of 999,9 ‰ minimum purity in a suitable form.

The content of each impurity shall be specified.

6 Apparatus

6.1 Customary laboratory apparatus.

6.2 ICP OES, with

— fixed and/or scanning channels,

— an optical resolution of 0,02 nm for the relevant elements and a detection limit of 0,05 mg/l or better, and

— the capability of background correction.

For preferably used wavelength, see Annex A.

6.3 Analytical balance, with a reading accuracy of 0,01 mg.

7 Procedure

WARNING — Suitable health and safety procedures should be followed.

7.1 Sample solution

For each sample to be analysed, two sample solutions shall be prepared as follows.

Weigh approximately 500 mg of the sample portion to the nearest 0,1 mg, transfer into a 50 ml volumetric flask, and add 30 ml of aqua regia (5.3). Heat gently until complete dissolution of the sample and continue to heat to expel the nitrogen oxides. Allow to cool, make up with water to 50 ml, and mix thoroughly.

The deviation in the volume of the volumetric flask caused by heating is acceptable for this International Standard.

If insolubles are observed, dissolution under pressure should be performed.

7.2 Calibration solutions

Weigh two portions of (500 ± 2,5) mg of the reference material (5.5) and dissolve each one as specified in 7.1.

Blank solution. Allow to cool and then make up with water to 50 ml and mix thoroughly.

Calibration solution. Allow to cool, add 5 ml of acid stock solution (5.4) or a volume corresponding to the expected amount of the trace element in the matrix to the second reference material solution, make up with water to 50 ml, and mix thoroughly.

7.3 Measurement

Set up the instrument in accordance with the manufacturer's instructions and choose appropriate background correction positions. A clean torch, spray chamber, and sample uptake tubes shall be used and the plasma shall be stabilized before use, following the recommendations of the instruments manufacturer.

Spray the calibration solutions 1 and 2 in accordance with the defined instrument calibration procedure and then run the analytical procedure for the sample solutions. The result shall be displayed with enough decimal places to provide an accurate indication of concentrations at the detection limits of the relevant elements.

Each solution shall have a stabilization time of at least 30 s, followed by five integrations of at least 5 s each for the determination of the net intensities (i.e. background-corrected).

The rinsing time between each measurement shall be sufficient to allow the signal to come back to the baseline, except for the matrix element.

The intensity of the chosen matrix line (see Tables A.1, A.2, and A.3) shall not be included in the calculation as described in 8.2.

8 Calculation and expression of the test results

8.1 Calibration curves

Set the concentration in the blank solution and the calibration solution, taking into account impurities introduced in the solution by the reference material (5.5) and calculate the calibration curve for each element using the net intensities obtained for the blank solution and the calibration solution.

8.2 Calculation

By means of the calibration curves (see 8.1), convert the net intensity values into concentration values and use Formula (1) to calculate the mass portion of each relevant element (W_i).

$$W_i = \frac{c_i \cdot V_s}{m_s} \quad (1)$$

where

c_i is the concentration of element i in the sample solution, in mg/l;

V_s is the volume of the sample solution, in litres;

m_s is the mass of the metallic sample, in milligrams.

The detection limit is defined as three standard deviations of the concentration of each individual element measured in the calibration solution.

The specific precious metal fineness, W_{sp}, in parts per thousand, is thus calculated using Formula (2).

$$W_{sp} = 1\,000 - \left(\sum W_i \cdot 1\,000\right) \quad (2)$$

where

$\sum W_i$ is the sum of the mass portion of each element found above its detection limit.

8.3 Repeatability

The results of duplicate determinations shall not deviate more than 0,1 ‰ of the precious metal fineness. If the variation is greater than this, the assay shall be repeated.

9 Test report

The test report shall include at least the following information:

a) identification of the sample including source, date of receipt, and form of sample;

b) sampling procedure;

c) the method used by reference to this International Standard, i.e. ISO 15093;